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45°/0° Reflectance Factors of Pressed Polytetrafluoroethylene (PTFE) Powder

P. Yvonne Barnes and Jack J. Hsia

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Radiometric Physics Division
Physics Laboratory
National Institute of Standards and Technology
Gaithersburg, MD 20899-0001

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P. Yvonne Barnes and Jack J. Hsia

Radiometric Physics Division, National Institute of Standards and Technology, Gaithersburg, MD 20899, USA

Abstract

Pressed polytetrafluoroethylene (PTFE) powder is used for 45°/0° reflectance factor standards. The radiometric and spectrophotometric measurement community such as the Council for Optical Radiation Measurements (CORM) has demonstrated the need for such a standard and its application to quality control and quality assessment. This publication briefly describes the instrumentation used for the 45°/0° reflectance factor measurements of pressed PTFE powder from 380 nm to 770 nm. Also, the variations of 45°/0° reflectance factor with sample preparation and materials are discussed. The expanded uncertainty at a coverage factor of two for the 45°/0° reflectance factors of pressed PTFE powder ranges from 0.009 to 0.017.

Keywords

Diffuse Reflectance, 45°/0° Reflectance Factor, Polytetrafluoroethylene resins, Pressed PTFE powder, PTFE, Reflectance Factor

1. INTRODUCTION

Over the years, the Radiometric Physics Division at the National Institute of Standards and Technology (NIST) has investigated [1,2] the reflection properties of polytetrafluoroethylene (PTFE) resins, in a form of granular molding powder. NIST has established the 6°/hemispherical reflectance factor scale of pressed PTFE powder [3] and provided general preparation information on pressing PTFE powder (Appendix A). Recently, standards organizations, government agencies, national laboratories, optical material industries, and instrument manufacturers have expressed the need for a 45°/0° reflectance factor standard in a report on critical problems and projected national needs in optical radiation measurement by the Council for Optical Radiation

Measurements [4]. Although these requirements span the ultraviolet, visible and near infrared spectral regions, most of the requests for calibration are 45°/0° reflectance factors in the visible wavelength region.

The reflectance factor of a sample is defined [5] as "the ratio of the radiant flux reflected in the directions delimited by the cone to that reflected in the same directions by a perfect reflecting diffuser identically irradiated." This cone is formed by the receiver limiting aperture and with apex at the center of the sample surface. A perfect reflecting diffuser is an ideal (no loss) lambertian (uniform in all directions) diffuser. The 45°/0° reflectance factor is the reflectance factor measured at 45° incident angle and normal viewing angle. The governing equations and the description of how the reflectance factor is measured are given in Reference 6 and a reprint is included in Appendix B. The reflectance factor measurement method (see app. B) using a step-down technique and a view factor calls for the measurements of the ratio of two fluxes and, in addition, some linear dimensions.

In order to develop the pressed PTFE powder as an intrinsic standard, studies of the major factors that affect the measured values were performed. This publication describes the sample pressing method and briefly described the 45°/0° reflectometer for reflectance factor measurements of the samples from 380 nm to 770 nm. Also, the variations of 45°/0° reflectance factor with sample preparation and materials are discussed. Finally, a table is presented for 45°/0° reflectance factors and their associated uncertainties of pressed PTFE powder from 380 nm to 770 nm at every 10 nm.

2. SAMPLE PRESSING METHOD

This section describes the method used to prepare a diffuse PTFE sample that is uniform in both appearance and density (approximately 1 g/cm^3). After practice and familiarity with the presser (see fig. 1) [7], a sample can be made quickly, frequently, and with consistent results. For the best results and ease of processing, 25 g of PTFE powder is placed in a blender with sharp blades. The PTFE powder is pulverized until it appears light, airy and powder-like.

Referring to Figure 1, the diameter of the poly(methyl methacrylat) plunger is 51 mm; the height of the funnel is 75 mm; and the receptacle is 10 mm in depth. The end area of the plunger is pre-treated by pressing it on some amount of PTFE powder to reduce the influence of hydrocarbons in the plunger material. The following steps are followed to press a sample: 1) the funnel and the receptacle are assembled; 2) 25 g of pulverized PTFE powder is scooped into the funnel; 3) the pre-treated plunger is inserted into the funnel and manually pressed downward until it stops; 4) the plunger is withdrawn slowly to overcome the suction created by the close fit of the funnel and the plunger; 5) the receptacle is separated from the funnel by loosening the set screws; 6) the pressed sample is then imprinted with a sheet of pre-treated sandpaper by hand with minimum pressure. Some general information on PTFE powder and preparation

is provided in Appendix A. The safety aspects in handling and use of PTFE powder are listed in Reference 8.

3. 45°/0° REFLECTOMETER

The overall instrument consists of a source system and a 45°/0° reflectometer [6,9,10]. The source system consists of a tungsten ribbon-filament lamp, mirrors, polarizer, predisperser, monochromator, and other mirrors. The source system provides a nearly collimated, polarized output with 10 nm band pass. The configuration of the 45°/0° reflectometer is shown in figure 2. Details of the receiver system are shown in figure 2 of Appendix B.

Referring to figure 2a in Appendix B, the incident beam illuminates at 45° from the sample normal an area 21 mm by 21 mm at the center of the sample (S). Due to the change of the optical arrangement of the source system after the publication of the paper in Appendix B, the larger light beam still provides a 10 nm band pass. The receiver system views the entire sample at 0° from the sample normal. In figure 2b of Appendix B, two 15 cm diameter averaging spheres (AS) are mounted together vertically and the photomultiplier (D) is attached to the upper sphere. A precision circular limiting aperture (LA) on the lower sphere subtends a half angle of 2.8° from the center of the sample. This double-averaging sphere detection arrangement provides a uniform receiver system for both of the required measurement geometries: a small light beam reflected from a specular black glass plate and a larger diffusely reflected light beam from a diffuse sample.

4. MEASUREMENTS

Due to the low signal levels associated with this type of bidirectional reflectance factor measurement, a step-down technique is used to reduce the required dynamic range for measurements and to reduce measurement uncertainty. A specular black glass plate with a regular (specular) reflectance value of about 0.04 is employed. The 22.5°/22.5° regular reflectance of the black glass is measured, using a specular reflectometer [11], with light polarized parallel and perpendicular to the plane of incidence at each wavelength. The flux of the pressed PTFE sample is then measured relative to the flux of the black glass plate with the 45°/0° reflectometer for each polarization state. This relative value of the pressed PTFE sample, the specular reflectance of the black glass plate and the projected solid angle of the receiver limiting aperture are used to calculate the 45°/0° reflectance factor according to Eq (9) in Appendix B.

The 45°/0° reflectance factors of pressed PTFE powder were performed on ten freshly pressed samples. These samples were prepared by one operator, taken from one batch of PTFE powder, and measured over the visible spectral range. The standard deviation for these measurements for a coverage factor (k) of 2 was less than 0.001

indicating a good repeatability. There was no spectral dependence. The Type B uncertainty [12] for k = 2 was 0.0027 for the visible spectral region [6]. The contributors to Type B uncertainty were receiver system non-linearity and non-uniformity, scattered flux, and angular setting and view factor uncertainties.

4.1 Operator Variability

A study was conducted to investigate variability in the results when ten different laboratories prepared samples. Each laboratory received an instruction sheet, a presser, a container with pulverized PTFE powder, and a pre-labeled box with packing material to return the pressed PTFE samples to NIST. Each laboratory pressed two samples with the proper amount of PTFE powder needed to prepare a sample of 1 g/cm^3 in density, 51 mm in diameter and 10 mm in thickness. No measurements were required from the laboratories and all twenty samples were returned to NIST for measurements. Results of this study are reported in Table 1, which shows the difference of the reflectance factor for each sample from the mean value of 20 samples at each of the five wavelengths. The Type A uncertainty (k = 2) of these differences for the 20 samples range from 0.006 to 0.012 in the wavelength region from 380 nm to 770 nm.

4.2 Material Variability

The variability in the results due to the composition of the PTFE powder was studied by making samples from four different drums of PTFE powder. The Ausimont[1], type G-80 and type Algoflon F5 PTFE powder were used in this investigation. Each of these powders was produced at a different time with different lot numbers. The samples were made as described above and measured at seven wavelengths. The quantity reflectance factor for each drum at the measurement wavelengths is shown in Table 2. The Type A uncertainties (k = 2) for measurements of four drums range from 0.013 to 0.001 for the wavelength range from 380 nm to 770 nm.

4.3 45°/0° Reflectance factor and uncertainty

The 45°/0° reflectance factors of pressed PTFE powder of samples from four drums in Table 2 were averaged at each of the seven wavelengths and these values were

1. Certain commercial instrument or materials are identified in this paper in order to specify adequately the procedure. In no case does such identification imply endorsement or evaluation by the National Institute of Standards and Technology.

interpolated by spline fit for other wavelengths. These values are reported in Table 3 together with the expanded uncertainties for a coverage factor of 2. These uncertainties include the type B uncertainty, statistical measurement uncertainty, operator to operator variations, and material to material variations. The standard deviations (k = 2) range from 0.017 to 0.009 for wavelengths from 380 nm to 770 nm.

5. REMARKS

The results of this investigation show that the expanded uncertainties of the 45°/0° reflectance factors of pressed PTFE powder mainly due to two factors, one is the material variability which is larger at the shorter wavelengths and the other is the operator variability. Furthermore, these expanded uncertainties are about twice those of the 6°/hemispherical reflectance measurements [2].

The following laboratories participated in the PTFE powder pressing study described in this publication: Biospherical Instruments, San Diego, CA; Byk-Gardner, Silver Spring, MD; Datacolor, Lawrenceville, NJ; Hughes, Danbury, CT; Hunter Lab, Reston, VA; Jet Propulsion Laboratory, Pasadena, CA; Labsphere, North Sutton, NH; Miles Lab, Elkart, IN; Munsell Color Science Laboratory, Rochester Institute of Technology, Rochester, NY; University of Arizona, Tuscon, AZ.

Both the 45°/0° reflectometer and the specular reflectometer used in this investigation were replaced in 1995 by a new Spectral Tri-function Automated Reference Reflectometer (STARR) incorporating new detectors and electronics. Preliminary intercomparisons between the old and new instruments on 45°/0° reflectance factor show measurements agreeing to within 0.002.

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Table 1. Operator Variability : Each laboratory returned two pressed PTFE samples; alpha-numerics indicate the laboratory (A - J) or the sample (1 - 2). The quantity reflectance factor minus the average for the twenty samples is shown for each sample at the measurement wavelengths. The Type A uncertainty (k = 2) for the 20 measurements is shown in the last row.

	Wavelength [nm]				
	380	450	600	700	770
A1	0.002	0.004	0.002	0.003	0.004
A2	0.004	0.006	0.003	0.001	0.003
B1	-0.001	0.001	0.000	0.000	-0.007
B2	-0.005	0.000	-0.001	0.000	-0.001
C1	0.009	0.006	0.004	0.004	0.005
C2	-0.005	-0.005	-0.004	-0.006	-0.005
D1	0.001	0.002	0.001	0.001	0.002
D2	0.007	0.004	0.003	0.002	0.002
E1	-0.003	0.002	0.002	0.003	0.004
E2	-0.004	-0.004	-0.005	-0.006	-0.006
F1	0.000	-0.011	-0.004	0.008	0.004
F2	-0.001	0.000	0.001	0.001	0.001
G1	-0.002	-0.004	-0.004	-0.004	-0.005
G2	0.009	0.005	0.002	0.002	0.004
H1	-0.012	-0.006	-0.006	-0.008	-0.007
H2	0.010	0.001	0.000	0.001	0.002
I1	-0.003	0.000	0.002	-0.001	0.000
I2	0.006	-0.002	0.002	0.002	0.005
J1	-0.010	-0.007	-0.005	-0.007	-0.004
J2	0.003	0.007	0.006	0.008	0.006
std dev (k = 2)	0.012	0.010	0.006	0.008	0.008

Table 2. Material Variability : Pressed PTFE samples from four drums, each drum was manufactured at a different time. The quantity reflectance factor is shown for each drum at the measurement wavelengths. The mean and Type A uncertainty (k = 2) for the measurements of the four drums are shown in the last two rows.

	Wavelength [nm]						
Drum #	380	420	470	550	630	700	770
1	1.005	1.008	1.011	1.013	1.012	1.013	1.016
2	1.001	1.006	1.009	1.011	1.012	1.012	1.017
3	0.993	0.999	1.003	1.007	1.010	1.011	1.016
4	1.007	1.010	1.011	1.012	1.013	1.013	1.017
mean	1.002	1.006	1.009	1.011	1.012	1.012	1.017
std dev (k=2)	0.013	0.010	0.008	0.005	0.002	0.002	0.001

Table 3. 45°/0° Reflectance Factors of Pressed PTFE Powder (1 g/cm^3)

Wavelength [nm]	Reflectance Factor	Uncertainty* k = 2
380	1.002	0.017
390	1.003	0.017
400	1.005	0.016
410	1.006	0.016
420	1.006	0.015
430	1.007	0.015
440	1.007	0.015
450	1.008	0.014
460	1.008	0.014
470	1.009	0.014
480	1.009	0.014
490	1.009	0.013
500	1.010	0.013
510	1.010	0.012
520	1.010	0.012
530	1.010	0.011
540	1.011	0.011
550	1.011	0.011
560	1.011	0.011
570	1.011	0.010
580	1.011	0.010
590	1.011	0.010
600	1.011	0.010
610	1.011	0.009
620	1.012	0.009
630	1.012	0.009
640	1.012	0.009
650	1.012	0.010
660	1.012	0.010
670	1.012	0.010
680	1.012	0.011
690	1.012	0.011
700	1.012	0.011
710	1.013	0.011
720	1.014	0.011
730	1.015	0.011
740	1.015	0.011
750	1.016	0.011
760	1.016	0.011
770	1.017	0.011

* The total uncertainty consists of the instrument uncertainty, material and operator variations.

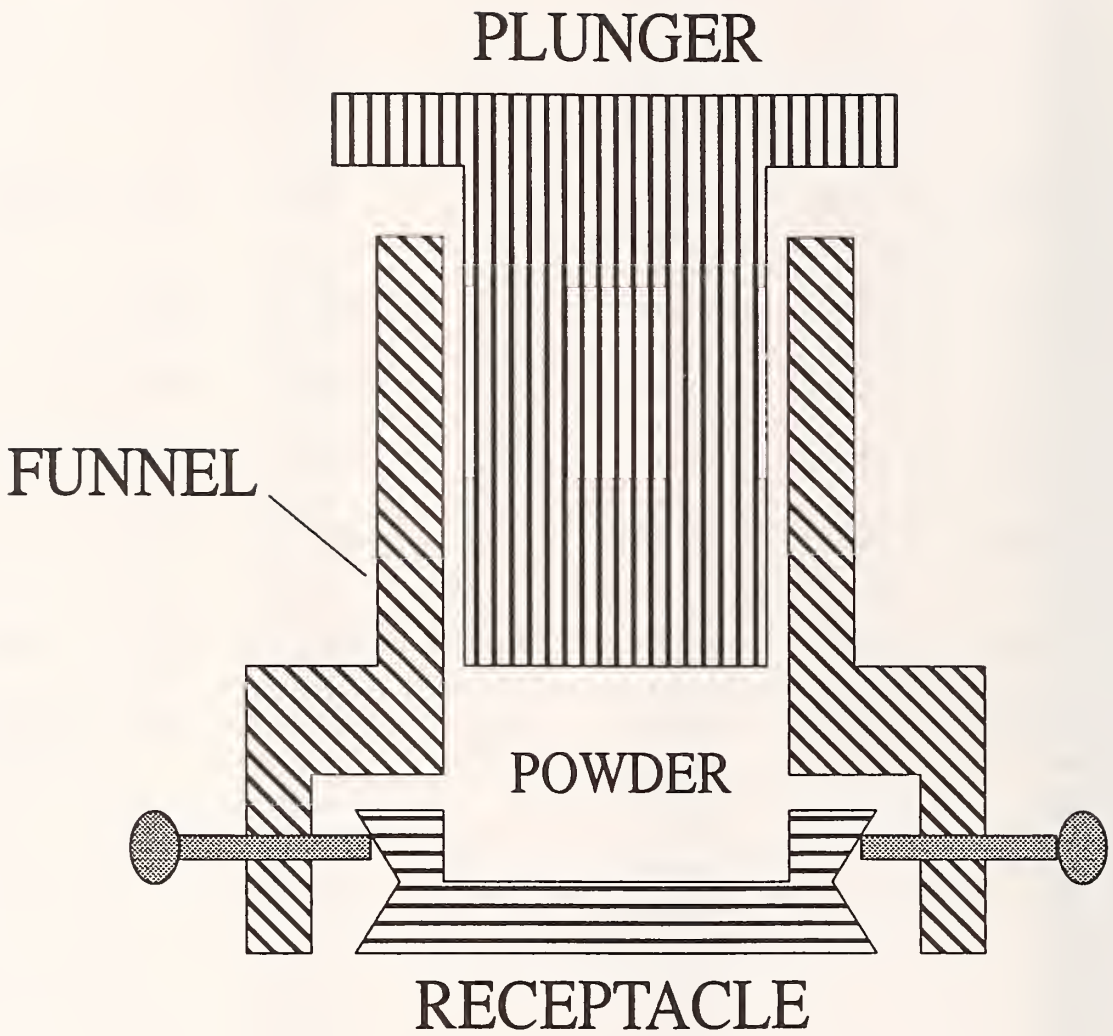

Figure 1. PTFE powder presser designed to produce a sample with spatial uniformity in density and in appearance.

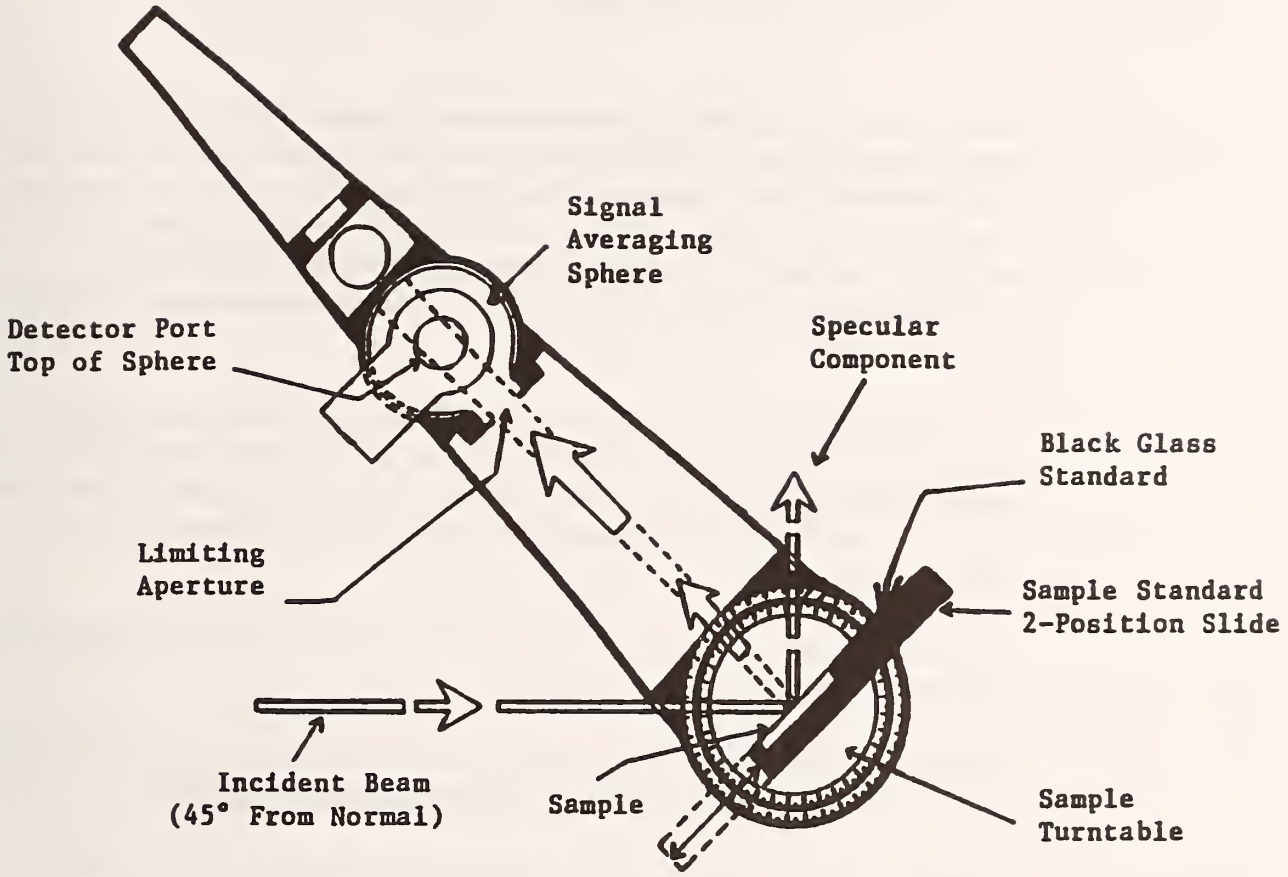

Figure 2. 45°/0° Reflectometer System
(Top View)

APPENDIX A. General and Advance Preparation Information for Pressing PTFE Samples

a. PTFE presser: Since there are no commercially available PTFE pressers that met our specifications, a simple PTFE presser was designed and fabricated at NIST (Refer to fig.1). The presser consists of three parts: a plunger that was constructed of poly(methyl methacrylate), a funnel of sufficient size to hold bulk PTFE powder before pressing, a receptacle designed to fit into the specimen holder of the 45°/0° reflectometer. This plunger was preconditioned by pressing against clean PTFE powder.

b. Commercial blender: PTFE powder naturally clumps together as aggregates. An Osterizer [13] blender was used to pulverize PTFE powder into a fluffy powder-like appearance. This type of commercial blender was chosen because it could be attached to glass jars and the blades could be sharpened in the machine shop.

c. Alternative devices: A stainless sifter or a flour sifter may be used instead of a commercial blender.

d. Static charge: PTFE powder acquires an electro-static charge throughout the pulverization process and will coat the glass blending jar and blades.

e. Utensils: Only stainless steel, glass, and porcelain materials or containers should be used to handle PTFE powder. The ultra-violet spectral reflectance factors of a pressed PTFE sample are affected by the hydrocarbons in plastic materials.

f. Containers of PTFE powder: PTFE powder is usually shipped by the manufacturer in drums of either 20 kg or 45.5 kg. The powder is packed in a plastic bag. Only the PTFE in the center portion of the drum is used, thus avoiding the powder that came in contact with the plastic bag.

g. Preconditioned sandpaper: A sheet of 150 grit size sandpaper was rubbed with a piece of sintered (in an electric furnace at 370 °C for 1 hour) PTFE, and shaken repeatedly so that the loose particles would fall off and not be embedded to the surface of the sample.

h. Ceramic tile: Use a ceramic tile or a similar type of object to evenly distribute the applied force over the preconditioned sandpaper on the sample area.

i. Laboratory conditions: PTFE powder is contaminated by airborne particles and smoke due to the electro-static charge (see sec. d above).

j. Safety: Obtain a Material Safety Data (MSD) sheet from the manufacturer of PTFE powder. Safety precautions and safety issues are the sole responsibility of users.

k. PTFE sample: A pressed sample of PTFE is durable and will not fall out of the receptacle. A sample may be positioned vertically, and if necessary it may also be inverted and tapped lightly so that the loose particles would fall off.

l. Cleaning. A clean camel-hair brush can be used to remove pieces of lint on the sample surface.

APPENDIX B. Reprint, NBS 45°/Normal Reflectometer for Absolute Reflectance Factors
(see next six pages)

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NBS 45°/Normal Reflectometer for Absolute Reflectance Factors

Jack J. Hsia and Victor R. Weidner

National Bureau of Standards, Washington, DC 20234, USA

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Abstract

A 45°/normal reflectometer has been constructed and tested for calibrating the absolute reflectance factor of diffuse samples over the 380–770 nm spectral range using polarized radiation. The measurement equations have been derived for the method used. The method using a step-down technique and view factor calls for the measurements of the ratio of two fluxes and, in addition, some linear dimensions. The uniformity of the receiver system is achieved by means of a double-sphere signal averager. Uncertainties of the absolute-reflectance-factor measurements obtained with this system are estimated to be ±0.3% of the measured value. For all the samples that have been tested, the 45°/normal reflectance factor was found to be higher than the 6°/hemispherical reflectance factor. The higher reflectance values for 45°/normal geometry were confirmed by additional gonioreflectometer measurements.

1. Introduction

Measurements of the diffuse reflecting characteristics of materials are of basic importance in radiometry, photometry and colorimetry as well as many other fields of science, technology and industry.

The International Commission on Illumination (Commission Internationale de l'Eclairage – CIE) recommended that starting in 1969, in colorimetric measurements of opaque materials, the perfect diffuser was to be taken as the primary reference standard. There is no existing material having diffuse reflection properties corresponding to the perfect diffuser with sufficient approximation. Therefore, the characteristics of all physical standards actually employed in comparison measurements must be established by techniques through which their absolute reflectance factors can be determined. The measurements involve not only the determination of the ratio of radiometric quantities but also the determination of linear dimensions.

The four geometries recommended by the CIE are 45°/normal, normal/45°, diffuse/normal, and normal/diffuse. The method and instrumentation to realize the NBS scale of directional-hemispherical reflectance factor has been described in [1].

For the 45°/normal and normal/45° reflectance factor measurements, there exists a large ratio of the incident flux to the reflected flux contained in a small solid angle. Various step-down techniques have been used by several National Laboratories [2–8] to insure sufficient accuracy over this wide dynamic range.

The purpose of this paper is to describe the method and the instrumentation used to establish the NBS scale of absolute reflectance factor in the 45°/normal geometry.

2. Measurement Equations

An absolute reflectance factor is defined [9, 10] as the ratio of the radiant flux reflected in the directions bounded by a given cone with the apex at a point of the surface under test to that reflected in the same directions by a perfect reflecting diffuser identically irradiated. The term "perfect reflecting diffuser" means the ideal (lossless) lambertian (uniform in all directions) diffuser. If the solid angle of the cone approaches zero, or 2π steradians, the reflectance factor approaches radiance factor or reflectance, respectively.

The reflectance factor of sample x, R_x, can be expressed as

$$R_x = \frac{\int^5 L(U,\lambda)\, S_x(U,u,\lambda)\, r(u,\lambda)\, U\cdot dA\; d\Omega u\cdot da\; d\omega\; d\lambda}{\int^5 L(U,\lambda)\, S_{id}\, r(u,\lambda)\, U\cdot dA\; d\Omega\; u\cdot da\; d\omega\; d\lambda} \qquad (1)$$

where L is the incident radiance; S_x and S_{id} are the scattering functions of the sample and of the ideal surface, r is the relative responsivity of the receiver; U and u are the direction vectors of travel of the incident and emergent radiation; $d\Omega$ and $d\omega$ are elements of solid angle oriented in the direction U and u; dA and da are elements of area through which the energy passes on to the sample and emerges from the sample; $d\lambda$ is the element of wavelength; and the symbol $\int^n$ indicates inte-

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gration with respect to n variables. In general, the nomenclature (especially the symbols) used here is that of reference [11]. There it is more completely defined and discussed.

If there is no interaction between the wavelength dependence and the geometrical dependence of the incident radiance and of the responsivity, the functions are separable. For any source function $C(\lambda)$ and luminous efficiency function $V(\lambda)$, Eq. (1) can be written as

$$R_x = \frac{\int C(\lambda)\, V(\lambda)}{\int C(\lambda)\, V(\lambda)} \qquad (2)$$

$$\frac{[\int^4 L(U)\, S_x(U,u,\lambda)\, r(u)\, U\cdot dA\, d\Omega\, u\cdot da\, d\omega]\, d\lambda}{[S_{id} \int^4 L(U)\, r(u)\, U\cdot dA\, d\Omega\, u\cdot da\, d\omega]\, d\lambda}$$

$$R_x = \frac{\int C(\lambda)\, V(\lambda)\, R_x(\lambda)\, d\lambda}{\int C(\lambda)\, V(\lambda)\, d\lambda} \qquad (3)$$

The spectral reflectance factor $R_x(\lambda)$ is expressed as

$$R_x(\lambda) = \frac{\int^4 L(U)\, S_x(U,u,\lambda)\, r(u)\, U\cdot dA\, d\Omega\, u\cdot da\, d\omega}{S_{id} \int L(U)\, r(u)\, U\cdot dA\, d\Omega\, u\cdot da\, d\omega} \qquad (4)$$

For a receiver system with uniform responsivity within the cone,

$$r(u) = r \text{ within } \omega \text{ and } r(u) = 0 \text{ outside of } \omega. \qquad (5)$$

The scattering function of a perfect diffuser could be written as

$$S_{id} = \delta(P - p)/\pi \qquad (6)$$

where P and p are position vectors on the testing surface for incident space and the emergent space, respectively.

Thus (4) can be expressed as

$$R_x(\lambda) = \frac{\pi}{\omega_p} \frac{\Phi_x(\lambda)}{\Phi_i(\lambda)} \qquad (7)$$

where $\omega_p = \int^2 u\cdot da\, d\omega$ is the projected solid angle of the cone onto the surface to be measured.

$\Phi_x = \int^4 L(U)\, S_x(U,u,\lambda)\, U\cdot dA\, d\Omega\, u\cdot da\, d\omega$ is the reflected radiant flux for the sample that enters the limiting receiver aperture, and

$\Phi_i = \int^2 L(U)\, \delta(P - p)\, U\cdot dA\, d\Omega$ is the incident radiant flux.

In radiation heat transfer [12, 13], ω_p/π (or f_{1-2}) is named the view factor (alternatively designated as the form factor, configuration factor, geometrical factor, or angle factor) representing the fraction of the radiant flux leaving a diffuse surface that is entering the limiting aperture. In other words, f_{1-2} is the fraction of the incident flux reflected from (1) a perfect diffuser that is entering the (2) limiting aperture. The spectral reflectance factor can now be expressed as

$$R_x(\lambda) = \frac{1}{f_{1-2}} \frac{\Phi_x(\lambda)}{\Phi_i(\lambda)} \qquad (8)$$

Equation (8) clearly indicates that the reflectance factor is the ratio of the reflected flux (into the limiting aperture) to that of a perfect diffuser. If another sample S with known reflectance is used to step down[1] the incident flux, the reflectance factor of sample x can be calculated as

$$R_x(\lambda) = \frac{1}{f_{1-2}} \frac{\Phi_x(\lambda)}{\Phi_S(\lambda)} \cdot \rho_S \qquad (9)$$

where Φ_S and ρ_S are the flux and the reflectance of the step-down sample S, respectively. The view factor will be expressed in detail below.

For a circular limiting aperture (diameter D) which is parallel to the sample surface at a distance d, and for $\Delta A1$, a small portion of the illuminated area, and the distance between a point in $\Delta A1$ and the central normal (on the sample surface) to the circular area of the limiting aperture being c, (see Fig. 1) the view factor can be expressed [14] as:

$$f_{\Delta A1-A2} = [1 - J/K]/2, \qquad (10)$$

where $J = 1 + G^2 - B^2$

$K = [G^4 + 2G^2(1 - B^2) + (1 + B^2)^2]^{1/2}$

$B = D/2d$

$G = c/d$

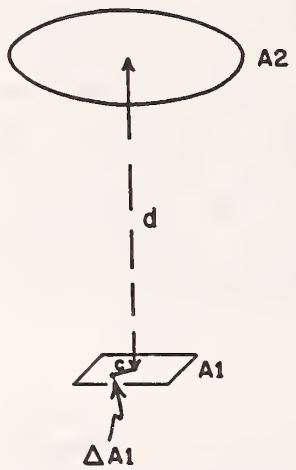

Fig. 1. Schematic for view factor (Eq. 10) Determination

When the illuminated area is very small and is at the central normal to the circular area of the limiting aperture, the view factor becomes

$$f_{1-2} = f_{\Delta A1-A2} = [1 + 4\, d^2/D^2]^{-1} \qquad (11)$$

and when the illuminated area is finite, the view factor is

$$f_{1-2} = \frac{1}{A1} \int f_{\Delta A1-A2}\, dA1 \qquad (12)$$

$$\text{or } f_{1-2} = \frac{\Delta A1}{A1} \sum_{A1} f_{\Delta A1-A2} \qquad (13)$$

if $\Delta A1$ is selected to be sufficiently small.

[1] Procedure described in Sect. 5

3. NBS Reference Spectrophotometer

The 45°/normal reflectometer is an accessory to the NBS reference spectrophotometer for reflectance [15]. The light source, monochromator, electronics and other associated equipment are located in a system control room. The exit slit housing of the monochromator is attached to a light-tight diaphragm in a wall, which allows the exit light beam or sample beam to enter a second room where the various reflectance-measuring devices, such as the specular reflectometer or the 45°/normal reflectometer, are installed. The advantages of this arrangement are that the second room can be used as an experimental dark chamber, while the light source, electronics and control systems are isolated from the experimental area. The sample beam emerging from the exit slit of the monochromator can be controlled to provide a spectral bandpass of 2, 5, 10, or 20 nm. For a 10-nm bandpass, the light beam has a cross section 10 by 18 mm at the sample position.

To measure spectral reflectance factor over the visible wavelength range of the monochromator, the system employs a tungsten strip lamp and a photomultiplier detector. A complete description of the spectrophotometer is given in an earlier publication [15].

Data have been published [16, 17] on the large effect of polarization on the reflectance factor. Therefore, the NBS design includes a polarization-insensitive receiver system and the measurements are made with polarized incident light. The final result is the average of measurements made with light polarized perpendicular and parallel to the plane of incidence.

4. 45°/Normal Reflectometer

The light beam from the source system is incident on a sample at 45° from normal and the reflected flux at normal to the sample is collected and measured. The schematic of the instrument is shown in Fig. 2. The 45°/normal rather than the normal/45° geometry was chosen, because the distance between the limiting aperture and the sample surface and, in tum, the view factor could be determined more accurately.

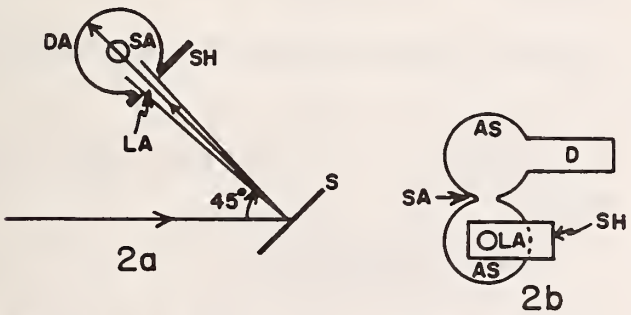

Fig. 2 a and b. Schematic of the 45°/normal Reflectometer. a Top view with top sphere not shown, for clarity. S: Sample, LA: Limiting Aperture, DA: Detector assembly, SA: Aperture between spheres; b Front view of the two-sphere Receiver Assembly, AS: Averaging Sphere, SH: Shutter, D: Detector, LA: Limiting Aperture, SA: Aperture between spheres

Because the step-down technique with a polished black glass is used to obtain the absolute reflectance factors of a diffuse sample, the instrument should be able to measure accurately both the specularly and the diffusely reflected light beams. This requires that the responsivity of the receiver system be uniform over its aperture area.

To fulfill the above requirement, the photomultiplier is equipped with a double-sphere signal averager. The spheres are approximately 15 cm in diameter. The sphere coating is a fluorocarbon powder, which is a nearly perfect diffuser of very high reflectivity. The two spheres are mounted together vertically and are designed to allow the inside curvature of the sphere coating to meet at the center of the port between the two spheres. The photomultiplier is mounted horizontally on the upper sphere. The spheres are attached to a stepping-motor-controlled vertical slide for alignment and scanning purposes. The lower sphere has a circular limiting aperture facing the sample. To prevent scattered radiation, other than that directly from the sample, reaching the limiting aperture, no component is placed between the surfaces of the sample and the limiting aperture. A black felt is placed on the top surface of the rotation arm to further reduce scattered radiation.

The sample holder, which can hold two samples, is mounted on a two-position slide that moves 12.5 cm between positions and is pneumatically driven. The slide is mounted on a stepping-motor-controlled turntable. The detector assembly is on an arm attached to another independently-controlled turntable having the same axis of rotation as that of the sample turntable.

The signal processing of the output from the photomultiplier utilizes a current-to-frequency converter that can integrate the detector output over a selected time interval, and a digital counter. The digital data from the counter are transferred to a computer for analysis.

5. Measurement Procedure

Before a measurement is performed, the instrument is first aligned. The sample holder can be adjusted normal to the incident light beam with the aid of a flat mirror in the sample position. The sample holder is then turned 22.5° from the normal position toward the limiting aperture. The limiting aperture is adjusted to center on the reflected light beam which is 45° from the incident light beam. A thin mirror, with the reflecting surface facing the limiting aperture, is held against the limiting aperture. The limiting aperture is aligned so that the light is reflected back to the exit slit of the monochromator. Thus the limiting aperture is aligned normal to the reflected light beam 45° from the incident beam.

The diameters of the limiting aperture were measured at eight places by using a shadow profile projector before the aperture was mounted on the lower sphere. The circularity has been checked by a deviation plotter. The average diameter is 38.238 mm. The distance be-

tween the surfaces of the limiting aperture and the sample is determined when the sample holder is turned 45° from the normal position. A thin glass plate is placed over the limiting aperture. The distance between this glass plate and a flat surface at the sample position is measured with an inside micrometer. The thickness of the glass plate is added to the micrometer measurement to give the total distance which in this case is 381.89 mm. The view factor can thus be calculated using (10) and (13).

The spectral 22.5° specular reflectances of a piece of polished black glass are first determined with the specular reflectometer [18] with light polarized perpendicular and parallel to the plane of incidence. This piece of black glass is then used to step down the incident flux to determine the 45°/0° reflectance factors of a sample. The flux of the sample in 45°/0° geometry is compared to the flux of the black glass in the 22.5°/22.5° geometry. The reflectance factor of the sample can thus be calculated using (9). The measurements are performed with polarized light, and the final result is the average of measurements made with light polarized perpendicular and parallel to the plane of incidence.

6. Performance

It is beyond the intent of this paper to describe in detail the various checks that were made on the performance of the monochromator, other components of the spectrophotometer, and the reflectometer, except to mention briefly the magnitudes of errors associated with this portion of the system, since they do influence the accuracy of measurements of reflectance factor. Information regarding some of these investigations is presented in [15].

Wavelength scale uncertainties, receiver linearity, receiver uniformity, scattered radiation, angular setting uncertainty, and view factor uncertainty are involved in checking the performance of the instrument.

The wavelength scale of the monochromator was checked by measuring the emission lines [18] of several line-source lamps and the instrument function, then determining the centroid wavelengths. Uncertainty in the wavelength scale is 1 nm or less for a 10 nm bandpass. Corrections for these errors are made by adjustment of the wavelength-scale setting to compensate for differences between the wavelength counter and the true wavelength.

The linearity of the receiver system was measured by the light addition method with a double aperture apparatus [20, 21]. The results indicate the receiver system is linear to better than ±0.1% and the nonlinearity is corrected for measured data.

Even though the receiver system utilizes double spheres as an averager device, there is still a slight nonuniformity causing 0.05% uncertainty in comparing the fluxes of specularly and diffusely reflected beams.

Radiation scattered off the components between the exit slit of the monochromator and the limiting aperture of the receiver system causes errors in a reflectometer. The room containing the reflectometer is lined with a black felt material to absorb scattered radiation. All mechanical components, including the sample holder and the limiting aperture, are black anodized to reduce the scattered radiation. Some interreflections between the limiting aperture and the sample and between the sphere coating and the sample are unavoidable. The scattered radiation caused by the interreflection is determined to be less than 0.05% of the reflected radiation.

The 0.05° uncertainty of the angular setting of the sample holder contributes to 0.15% uncertainty in determining the ratio of the reflected fluxes of the sample and the piece of black glass.

The uncertainties in determining the diameter of the limiting aperture, the distance between the sample surface and the limiting aperture, and the dimensions of the light image size on the sample surface result in an uncertainty of about 0.05% for the view factor.

An analysis of the various sources of error that may affect the accuracy of reflectance factor measurements on the NBS 45°/normal reflectometer indicates that the final uncertainty in these measurements will be of the order of ±0.3% of the measured value.

7. Ratio of R(45°/Normal) TO R(6°/Hemispherical) with Specular Component Excluded

The hemispherical reflectance factor R(0°/h) of a diffuser for normally incident light can be calculated from the angular variations of reflectance factors R(0°/θ) using the following expression:

$$R(0°/h) = \frac{\int_0^{\pi/2} R(0°/\theta) \cos\theta \sin\theta \, 2\pi d\theta}{\int_0^{\pi/2} \cos\theta \sin\theta \, 2\pi d\theta} \qquad (14)$$

Divided by R(0°/45°), Eq. (14) can be written as:

$$\frac{R(0°/h)}{R(0°/45°)} = 2 \int_0^{\pi/2} \frac{R(0°/\theta)}{R(0°/45°)} \sin\theta \cos\theta d\theta \qquad (15)$$

In terms of measurable quantities, (15) can be rewritten as:

$$\frac{R(0°/h)}{R(0°/45°)} = 2 \int_0^{\pi/2} B(0°/\theta) \sin\theta \cos\theta d\theta \qquad (16)$$

where

$$B(0°/\theta) = \frac{\Phi(0°/\theta)/\cos\theta}{\Phi(0°/45°)/\cos 45°} \qquad (17)$$

with Φ as the reflected flux from the sample.

The two working standards, vitrolite glass and porcelain enamel plate, one pressed $BaSO_4$ sample, and one pressed fluorocarbon sample were measured on the gonioreflectometer at 550 nm with 10 nm bandpass. The incident angle was 0° and the angles of viewing were from 5° to 85° at 10° intervals. Measurements were

made with light polarized parallel and perpendicular to the plane of incidence. $B(0°/\theta)$'s were calculated for each polarization and the averages obtained.

Average $B(0°/\theta)$'s at the discrete intervals were fitted by the least-squares method. Individual data did not deviate from the fitted curve by more than ±0.001 and were typically within 0.0005 of the fitted curve. The curve fittings were done by using a six-parameter equation:

$$B(0°/\theta) = \sum_{i=0}^{5} b_i \theta^i \tag{18}$$

Inserting (18) into (16), we have

$$\frac{R(0°/h)}{R(0°/45°)} = 2 \sum_{i=0}^{5} b_i I_i \tag{19}$$

where

$$I_i = \int_0^{\pi/2} \theta^i \sin\theta \cos\theta \, d\theta \tag{20}$$

The values [1] of the integrals I_i are (from i = 0 to 5): 0.50000, 0.39270, 0.36685, 0.37990, 0.42147, and 0.49129. With the values of I_i and b_i, $R(0°/h)/R(0°/45°)$ can be predicted using (19).

Six deg.-hemispherical reflectance factors, $R(6°/h)$, with specular component excluded and 45°/normal reflectance factors, $R(45°/0°)$, were also measured for the four samples. $R(6°/h)$ will not be different significantly from $R(0°/h)$, and $R(45°/0°)$ is equal to $R(0°/45°)$ according to the Helmholtz reciprocity principle.

The measured values of $R(45°/0°)$, $R(6°/h)$ and the ratio of the measured values and integrated ratios are given in Table 1. The measured and integrated values of the ratios agree to 0.005. These ratios are consistent with references [4, 22, 23] in that the values of 45°/normal reflectance factors are higher than the values of the 6°/h reflectance factors. And in the cases of vitrolite and enamel samples the ratios are about four percent higher than unity.

Table 1. Comparison of $R(45°/0°)$ and $R(6°/h)$* (at 550 nm)

Sample	Measured Value $R(45°/0°)$ (b)	$R(6°/h)$* (a)	$R(45°/0°)/R(6°/h)$ (b)/(a)	Integrated**
$BaSO_4$	0.987	0.982	1.005	1.002
Fluorocarbon	1.009	0.994	1.015	1.011
Vitrolite	0.922	0.884	1.043	1.044
Enamel	0.875	0.839	1.043	1.040

* Specular component excluded
** Integration using normalized gonioreflectance data

8. Summary

A 45°/normal reflectometer spectrophotometer was constructed for calibrating the absolute reflectance factor of diffuse samples over the 380–770 nm spectral range. The measurement equations were derived and the measurements were made as a function of wavelength with a polarized light beam. The system performance was examined for such parameters as wavelength scale uncertainties, receiver system linearity and uniformity, scattered radiation, angular setting and view factor uncertainties. Uncertainty in the measurements of reflectance factor will be of the order of ±0.3% of the measured value. Measured ratios of the 45°/normal reflectance factor to the hemispherical reflectance factor with specular component excluded were found to be greater than one for all the samples that have been tested. The higher reflectance values for 45°/normal geometry were confirmed by additional gonioreflectometer measurements.

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